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A PLANT FOR IMMOBILIZING LOW LEVEL LIQUID WASTE IN WATER EXTENDIBLE POLYMER (PART 2)

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ABSTRACT

A prototype plant for immobilizing low level decontamination wastes in water extendible polymer was fully commissioned. The plant was used to immobilize a number of simulated inactive decontamination wastes. In general, conditions of neutral or close to neutral pH, a waste to resin volume ratio of unity and catalyst concentration up to 3% led to a uniformly cured solid waste form. When the waste forms were scaled up to 100 L in volume, temperature gradients as high 60°C between the centre and the periphery of the solid were observed. The associated thermal stresses led to the formation of cracks within the solid.

job	file	date	report no.
32-500-100	837.83	April 13, 1988	88-44-K

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EXECUTIVE SUMMARY

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A. Husain
Engineer - Materials Chemistry Section
Chemical Research Department

Aqueous low level radioactive wastes at the nuclear stations arise from routine decontaminations of equipment. Solutions for which the 'decay, dilute and disperse' philosophy is not appropriate have to be immobilized before they can be stored at BNPDS. Previous investigations using water extendible polyester (WEP) as an immobilization agent have demonstrated that a satisfactory waste form with good leaching resistance is obtained. This immobilization technique will be the basis for solidifying low level aqueous wastes at Darlington NGS. The solid waste form would then be transported to BNPDS for storage and possible volume reduction by incineration.

The immobilization process involves the formation of an emulsion between the aqueous waste and organic polymer and subsequent solidification of the emulsion by addition of a suitable catalyst/promoter combination. Waste concentrations as high as 700 g/kg product can be obtained with the liquid waste being sealed in the closed cell structure of the cured composite.

A prototype plant for immobilizing the low level wastes was fully commissioned. In the plant, emulsion is produced by metering the waste solution and polyester resin to a high speed mixing emulsifying chamber. The emulsion continuously discharges into a mechanical line blender where-in a metered catalyst stream is blended with it. The catalysed emulsion is then collected into product containers. The plant was used to immobilize a number of simulated inactive decontamination wastes. In general, conditions of neutral or close to neutral pH, a waste to resin volume ratio of unity and catalyst concentration up to 3% led to a uniformly cured solid waste form. When the waste forms were scaled up to 100L in volume, temperature gradients as high as 60°C between the centre and the periphery of the solid were observed. The associated thermal stresses led to the formation of cracks within the solid.

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It was concluded that the WEP plant with some minor modifications would be suitable for field use. Additional experience in the preparation of large waste forms is, however, strongly recommended. Assessment of possible complications due to processing of active waste solutions and the extent of secondary waste arisings is also needed.

TABLE OF CONTENTS

		Page
ABSTRACT		
EXECUTIVE SUMMARY		
1.0	INTRODUCTION	1
2.0	PROCESS FLOW SHEET	3
3.0	IMMOBILIZATION SYSTEM CONTROL	3
4.0	COMMISSIONING DIFFICULTIES AND THEIR RESOLUTION	5
5.0	RECOMMENDED SYSTEM CHANGES	5
6.0	IMMOBILIZATION OF SIMULATED DECONTAMINATION WASTES	6
7.0	PREPARATION OF LARGE WASTE FORMS	10
8.0	CONCLUSIONS AND RECOMMENDATIONS	15
9.0	ACKNOWLEDGEMENTS	17
REFERENCES		18
APPENDIX		19

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To Dr. D. Mills
Director, Research Division

A PLANT FOR IMMOBILIZING LOW LEVEL
LIQUID WASTE IN WATER EXTENDIBLE POLYMER (PART 2)

1.0 INTRODUCTION AND BACKGROUND

Aqueous low level radioactive wastes at the nuclear stations arise from routine decontaminations of equipment. Solutions for which the 'decay, dilute and disperse' philosophy is not appropriate have to be immobilized before they can be stored at BNPDS. The accumulated volume of such solutions from all the nuclear stations presently totals 2 m^3 while future arisings are expected to be about 5 m^3 (1 m^3 for each four-unit station) annually/1/. Previous investigations/2-6/ using water extendible polyester (WEP) as an immobilization agent have demonstrated that a satisfactory waste form with good leaching resistance is obtained. This immobilization technique will be the basis for solidifying low level aqueous wastes at Darlington NGS. The solid waste form would then be transported to BNPDS for storage and possible volume reduction by incineration.

The immobilization process involves the formation of an emulsion between the aqueous waste and organic polymer and subsequent solidification of the emulsion by addition of a suitable catalyst/promoter combination. Waste concentrations as high as 700 g/kg product can be obtained with the liquid waste being sealed in the closed cell structure of the cured composite.

Krochmalnek/7/ has described in detail the design considerations for a WEP system to immobilize low level radioactive waste at Darlington. This has formed the basis for the procurement, assembly and operation of a full scale prototype aqueous waste solidification system at Research Division. The system is semi-automated and is operated from a local control panel. Alternately, it may be operated in a manual mode.

The prototype plant and its manual commissioning was described in an earlier report/8/. This report concludes the activities undertaken to make the WEP plant fully operational, i.e., in its semi-automated mode. Experience in immobilizing simulated inactive decontamination wastes using the WEP plant is described. This includes the production of waste forms up to 100L in volume.

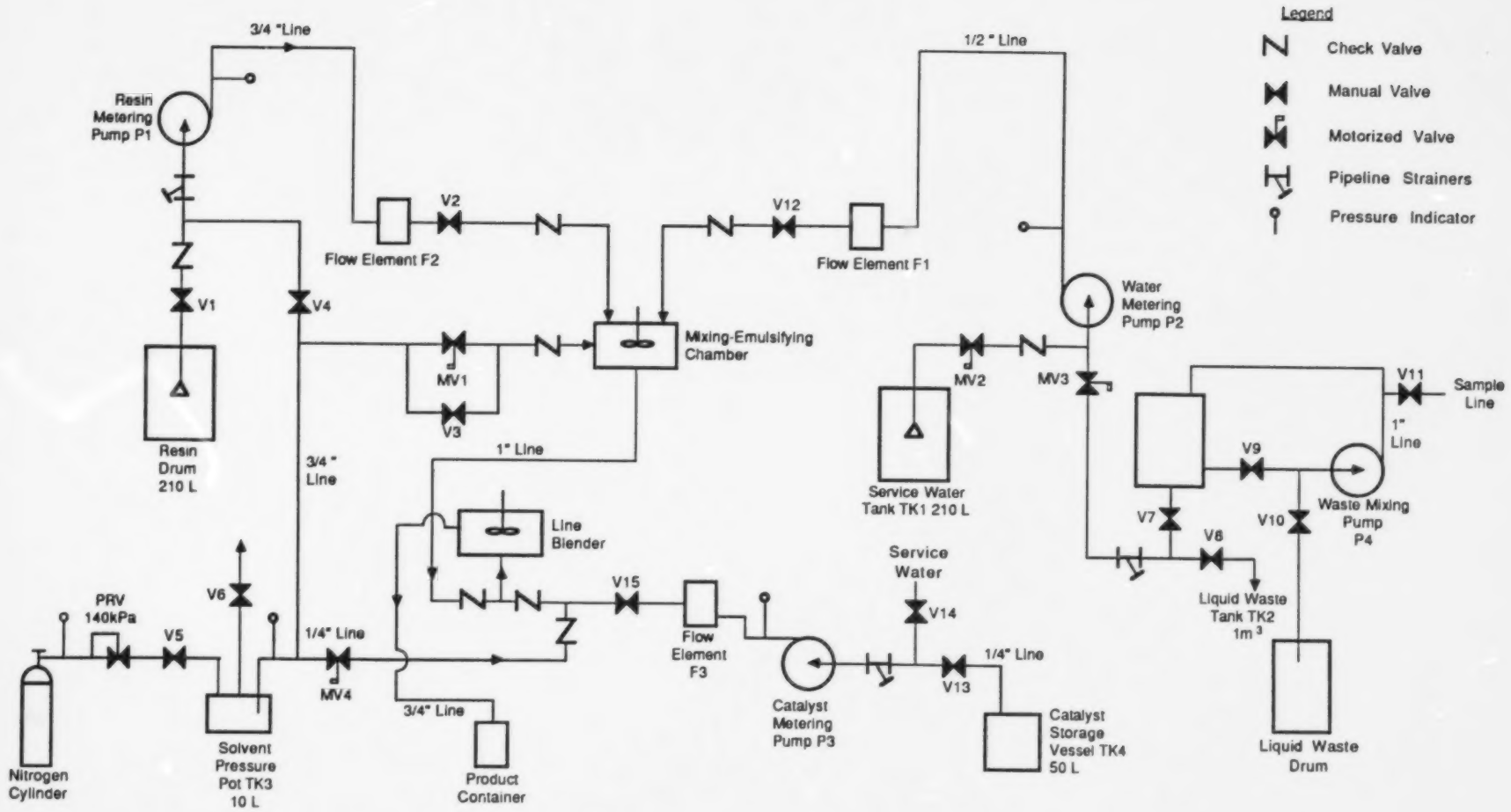

Figure 1. Flow Sheet of the WEP Immobilization Plant

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2.0 PROCESS FLOW SHEET

The flow sheet for the WEP system is shown in Figure 1. Waste solution from tank TK2 conditioned to pH 7-8 and the WEP resin (Ashland Chemical Co, Aropol WEP 661-P) from the resin drum are metered at 0.08 to 0.27 m^3/h to a mixing-emulsifying chamber to produce a water-in-resin emulsion. The catalyst, methyl-ethyl-ketone peroxide (Lucidol Lupersol DSW-9) is metered at 0.001 to 0.01 m^3/h and blended with the emulsion in the mechanical line blender. The catalyzed emulsion continuously discharges from the line blender into a product container. Provisions have been made (1) to permit liquid waste to be pumped from a drum into tank TK2 and the subsequent mixing and pH adjustment of its contents and (2) for service water/solvent flush of the emulsifying chamber and line blender at the end of an immobilization.

3.0 IMMOBILIZATION SYSTEM CONTROL

The production of catalyzed emulsion in the WEP plant may be performed either in the MANUAL or AUTOMATIC mode. Other functions i.e., transfer of waste from a drum into tank TK2, circulation of the contents of tank TK2 and solvent flush of the system are performed in the FILL, RECIRCULATE and FLUSH modes, respectively.

The control strategy for the semi-automated operation of the immobilization system has been described in detail elsewhere/7/. The main control system is depicted in Figure 2. Briefly, the 4 to 20 mA signals from the three flowmeters are added in accordance with appropriate scaling factors to obtain the composite 4 to 20 mA signal corresponding to the total flow rate entering the product container. When this signal, converted to a voltage pulse and accumulated over time, corresponds to the volume of the product container (see relationship between flow rate, time and integrator setting in Appendix), control action is taken to shut down the metering pumps. Following replacement of the filled container, operations are resumed by activating the pumps using a pushbutton control which automatically resets the integrator setting to zero and sequences the start up of the metering pumps. A manually set secondary control timer is used to protect against container overfilling should the flow totalizer malfunction.

Other control features include:

1. The pumps P1, P2 and P3 are interlocked to start (see Appendix) and stop together;

2. A zero flow condition in any (resin, waste or catalyst) line leads to system shutdown (see Appendix);

3. When the level in the tank TK2 falls below the lower level (non-intrusive) sensor on the tank, the pumps P2 and P4 automatically shutdown;

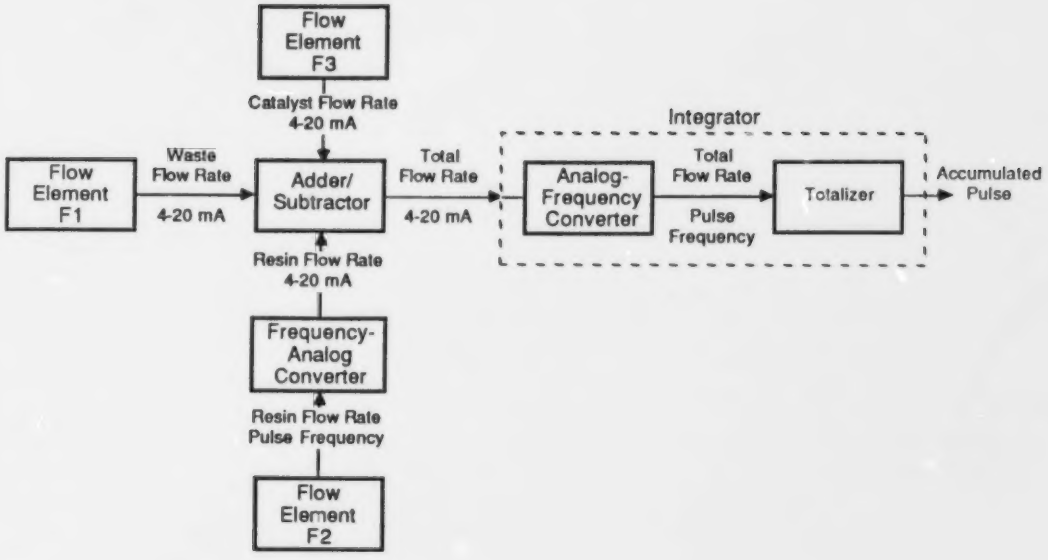


Figure 2. Block Diagram of the Main Control System

4. When the tank TK2 is filled above the higher level (non-intrusive) sensor, the pump P4 automatically shuts down the system;

5. Rotation sensors on the line blender and emulsion head shafts cause system shutdown if no motion is detected.

These control features were individually assessed during commissioning trials to ensure their satisfactory performance.

4.0 COMMISSIONING DIFFICULTIES AND THEIR RESOLUTION

The major problems encountered during the commissioning of the WEP plant are listed below. These are in addition to those described in the earlier report/8/.

1. Replacement of an empty resin drum with a fresh one was found to be a better practice than adding make-up resin to the same empty drum. The latter led to an accumulation of polymerized residues in the drum which caused chronic plugging of the inline resin strainer.

2. A vent above the mechanical seal in the line blender has to be kept open during its operation. Its inadvertent closure led to seal deterioration and eventual loss of the blender's function. The refurbished blender has to date operated faultlessly (with vent open of course).

3. Fluctuations in the catalyst line flow were experienced repeatedly. They inexplicably disappeared in the last stages of this work. Their cause could unfortunately not be identified.

4. Difficulties were frequently experienced in priming the resin metering pump even though it is supposed to be self-priming. Occasionally service water was used in the solvent pot for priming the resin pump. This caused rust formation in the pot. Blockage of the screen in the pot's intake line led to loss of solvent flow during an immobilization run. Subsequently, the solvent pot was periodically maintained to prevent a re-occurrence of this problem.

5.0 RECOMMENDED SYSTEM CHANGES

Recommended plant modifications based on the operating experience gained are listed below:

1. The 0.05 HP motor on the line blender is inadequate for blending catalyst in emulsions with viscosities considerably in excess of 1000 cps. It should be replaced with a heavier duty motor.

2. The solvent pot should be replaced with one of higher capacity and be equipped with a level gauge. This will decrease the make-up frequency.

3. In the AUTOMATIC mode, it is not possible presently to operate the system with the motorized valve MV2 open instead of MV3. Therefore, to immobilize residual (at the end of a run) waste in the system using a service water flush requires operation in the manual mode. Thereafter, in this mode with resin flow terminated and catalyst replaced with service water flow, the system is flushed clean of emulsion and catalyst. This step is then followed by a solvent flush in the FLUSH mode. It would be simpler conceptually to incorporate the service water immobilization step in the AUTOMATIC mode and to perform both water and solvent flush in the FLUSH mode. This would require the valves V13 and V14 to be motorized. The service water for the catalyst line flush may be drawn from the service water tank TK1.

4. The valves V2, V12 and V15 should be motorized.

6.0 IMMOBILIZATION OF SIMULATED DECONTAMINATION WASTES

Simulated decontamination wastes were prepared in accordance with the compositions specified in Table 1. The inclusion of detergent containing wastes in this and earlier studies is due to the possibility that these also may require immobilized storage as a result of unacceptably high activity levels for treatment through the generating station's Active Liquid Waste Treatment System. Immobilization of these wastes was performed at varying aqueous phase pH (in the range 4 to 10), waste-to-resin volume ratios (1.0 - 1.5) and catalyst concentrations (up to 6%). Conditioning of the waste involved addition of an appropriate acid or base. The base used was sodium hydroxide; it was added principally in the solid form to minimize increase in waste volumes.

The consistency of the emulsion (the line blender is capable of handling fluids with viscosities up to 1000 cps) and the rapidity of its cure are important considerations for operating the WEP plant. For these reasons and to confirm the results of earlier studies/1-4/, bench scale immobilizations were initially performed for each waste type. Based on their outcome immobilizations using the WEP plant were performed for some of the wastes.

Conditions under which a satisfactory immobilized product was obtained are summarized in Table 2 for each waste type. The acid/base conditioning requirements listed in the Table represent approximate amounts only. Due to a considerable variation in cure times for the various emulsified wastes, monitoring the progress of their cure was not consistent. Hence the cure times with few exceptions are reported as a range of values.

TABLE 1

CHARACTERISTICS OF DECONTAMINATION SOLUTIONS

NAME	COMPOSITION (wt %)	CONCENTRATION (g/kg)	pH
Turco 4512	Phosphoric acid	50	1.4
Turco 4521A	Ammonium citrate 66% Oxalic acid 34%	50	4.0
Turco 4306	Sulphamic acid	50	0.5
Calgon	Sodium hexametaphosphate	10	7.1
Nacconal	Detergent	10	7.7
Pickering solution	Sodium hexametaphosphate 50% Detergent 25% EDTA 25%	10	7.1
STPP	Sodium tripolyphosphate	10	8.5
Peroxide bicarbonate	Sodium carbonate 57% Sodium bicarbonate 27% Hydrogen perioxide 16%	127	9.6
Alkaline permanganate	Sodium hydroxide 77% Potassium permanganate 23%	130	13.0
Pèroxcit	Oxalic acid 54% Citric Acid 33% Hydrogen peroxide 13%	93	1.0

TABLE 2

IMMOBILIZATION OF SIMULATED DECONTAMINATION WASTES

	CONDITIONS				
NAME	pH	WASTE/ RESIN RATIO	% CATALYST	CONDITIONING REQUIREMENTS ACID/BASE PER L WASTE	CURE TIME
Turco 4512(*)	7.0	1.0,1.5	3.0-6.0	28 g NaOH	<24 h
Turco 4521A	7.0	1.0	1.0-3.0	10 g NaOH	2-4 h
		1.25	3.0-4.5	10 g NaOH	2-4 h
		1.5	4.5-6.0	10 g NaOH	2-4 h
	8.0	1.0	3.0	10 g NaOH	2-4 h
		1.5	3.0	10 g NaOH	>4 h
Turco 4306(*)	7.0	1.0	1.0-3.0	20 g NaOH	20 min
		1.5	3.0	20 g NaOH	45 min
Calgon	7.1	1.0	1.0-3.0	-	<50 min
		1.5	4.5-6.0	-	1<t<19 h
Nacconal+(*)	7.7	1.0	3.0	-	-
Pickering Solution	7.1	1.0	1.0-3.0	-	<15 min
		1.25	3.0	-	3-5 h
		1.5	4.5-6.0	-	3-5 h
STPP	8.5	1.0	1.0-3.0	-	>3 h
	7.0	1.0	3.0	1.3 ml conc. HNO_3	>24 h
Alkaline(*)	10	1.0	4.5-6.0	204 g sulphamic acid	1-3 h
Permanganate	7	1.0	4.5-6.0	212 g sulphamic acid	1-2 h
Peroxcit(*)	7	1.0	3.0	44 g NaOH	<2 h
	7	1.5	3.0	44 g NaOH	<24 h

* WEP plant immobilizations were not performed.

\+ Solutions contained 5 g/kg detergent.

The results indicate that in general, conditions of neutral or close to neutral pH, a waste to resin volume ratio (W/R) of unity and catalyst concentration up to 3% led to a uniformly cured solid waste form. These conditions are similar to those established in earlier laboratory studies /1-4/ indicating an insensitivity of the immobilization process to the intensity of agitation in the emulsification step. In general, an increase in W/R from 1.0 to 1.5 with catalyst levels up to 3% led to stable emulsions but inadequate waste forms possibly due to increased catalyst consumption by the chemicals in the waste. Catalyst levels up to 6% were required in these cases to yield a satisfactory waste form.

Unstable emulsions as in the case of 10 g/kg Nacconal solutions were due to an excessive waste loading. Dilution of the solutions to 5 g/kg yielded stable emulsions at a W/R ratio of unity and a satisfactory waste form using 1-3% catayst.

Conditioning of AP solutions was also attempted using oxalic and phosphoric acids/9/. However, the reaction with oxalic acid was rather violent and accompanied with considerable precipitate formation. In contrast, conditioning with phosphoric and sulphamic acids was readily accomplished with no adverse effects. Although AP solutions conditioned with phosphoric acid yielded stable emulsions, they could not be solidified even with catalyst concentrations as high as 6%.

Peroxide bicarbonate solutions formed extremely viscous emulsions at W/R ratios of 1.0 and 1.5. Immobilization of such emulsions using the WEP plant was impractical due to severe strain on the equipment, poor distribution of catalyst in the emulsion and inadequate heat dissipation in the solidifying waste form. Only a W/R ratio of less than unity would be practical for this waste type. Viscosity measurements suggested that an emulsion containing 40% waste conditioned to pH 8.5 (17 mL conc. H_3PO_4 per L waste) could possibly be immobilized using the WEP plant.

In addition to the solutions listed in Table 1, immobilization of a heat exchanger cleaning solvent containing 4% formic acid, 2% citric acid and 0.5% Rodine 31 was also attempted. The solvent is capable of dissolving calcium (as $CaCO_3$) and iron contained in the shell side deposits of Incoloy-800 tubed PNGS moderator heat exchangers/10/. The pH of the formulation is 1.85 and increases during application as calcium and iron are dissolved into it. Appropriately, therefore, the solvent was conditioned using $CaCO_3$. However, at pH 4 corresponding to 50 g/L $CaCO_3$, the solubility limit of the solution was exceeded and NH_4OH was used for further conditioning to pH 7 (required 21 mL/L). WEP emulsions with W/R ratios of 1.0 and 1.5 cured satisfactorily within 24 h using 3% catalyst. For a better simulation of the waste solution the solvent with added $CaCO_3$ (50 g/L) and Fe_2O_3 (10 g/L) was heated at 85°C for 6 h, cooled and filtered. The filtered solution contained 4.8% formic acid, 1.6% citric acid, 1.6% Ca and 30 ppm Fe (Rodine content was not measured) and had a pH of 3.6. It was conditioned with NH_4OH to pH values in the range 5-9. These yielded satisfactory solids at a W/R ratio of unity and catalyst level of 3%.

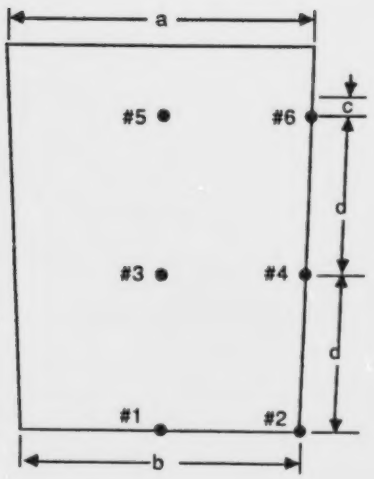
7.0 PREPARATION OF LARGE WASTE FORMS

The immobilization experience described earlier was based on observations of relatively small (not exceeding 500 mL) waste forms. In practice, a drummed waste product may have a volume of 200 L or greater and a diameter up to 100 cm. Due to its low thermal conductivity, poor heat dissipation during the cure of a large waste form may lead to product flaws not evident in smaller waste forms. To address this concern, several 20 L and one 100 L specimens were prepared in polyethylene product containers. Two of the specimens were prepared with thermocouples positioned as shown in Figure 3; one of these, a 20 L specimen was produced using a water/resin ratio of unity and 1% catayst (solidification time <10 min) while the second, the 100 L specimen was obtained using a calgon solution/resin ratio of unity and 4% catalyst (the higher catalyst level was chosen to yield a solidification time of ~30 min appropriate for a plant demonstration to client groups).

Figure 4 indicates the temperature history during the cure of the 20 L specimen. The temperature measurement at Position #6 (TC#6) was erroneus and is not reported. Due to the short container filling time, a temperature increase was experienced at all thermocouple locations almost at the same time (within 3-4 mins). The product appeared to have cured within 10 min of filling although the temperatures continued to rise well beyond this time. The maximum temperature recorded within the sample was 92°C at TC#3. The product cooled very slowly; this is particularly evident at its centre. Despite the large temperature gradients, the product was, however, free from any surface defects.

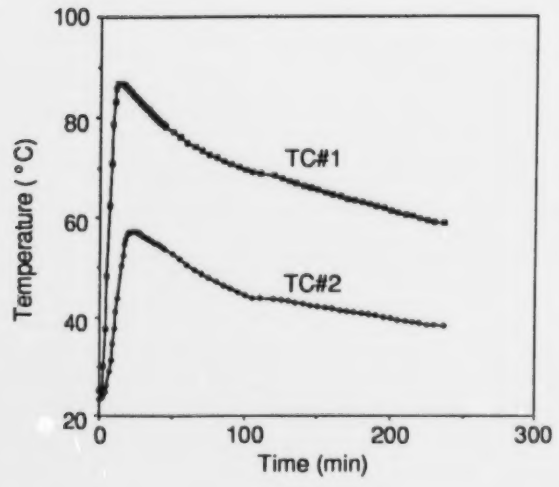
The temperature history during the cure of the 100 L specimen is shown in Figures 5 and 6. The relative displacement of the initial segment of the temperature curves in Figure 5 is due to the time, 10 and 23 min respectively, required to fill the product container to Positions 3 and 5. The maximum temperature recorded on the axial plane was ~107°C at all three positions. As with the 20 L specimen, the maximum temperatures occurred well beyond the duration (~30 min) required for solidification. The maximum temperature differences between the various thermocouple positions in the solid (cooling segment of the temperature curves) varied up to 60°C; the latter was measured between Positions 3 and 4. As before, the product cooled very slowly; at the centre (TC#3) the temperature even after 44 h measured as high as 52°C.

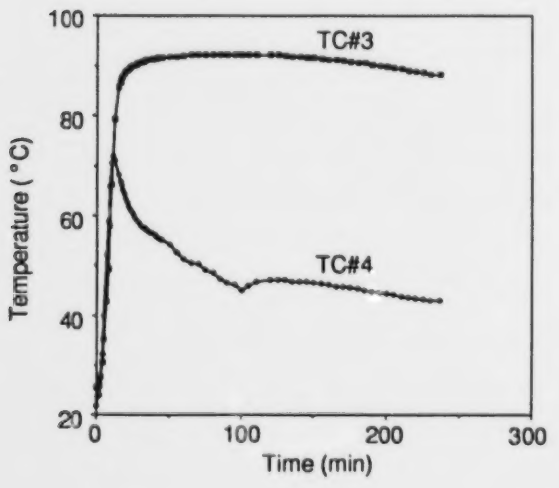
Examination of the 100 L waste form revealed that during the cure an air gap of 1.5 - 3 mm had developed round the container due to product shrinkage. This gap would undoubtedly have contributed to the difficulty in heat dissipation from the solid.



	Dimensions (cm)	
	20 L Specimen	100 L Specimen
a	29	53
b	26	48
c	1.5	5
d	15	15

Figure 3. Waste Form Dimensions and Thermocouple Positions





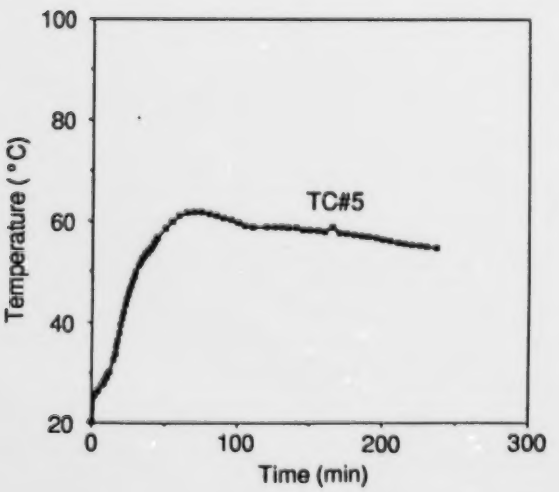


Figure 4. Temperature History During the Cure of a 20 L Specimen (see text for details)

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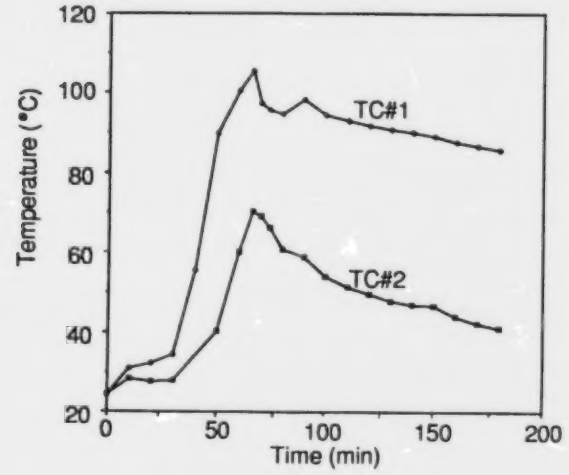

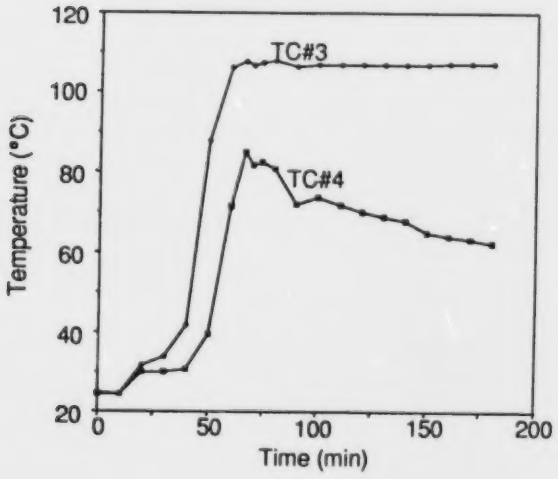

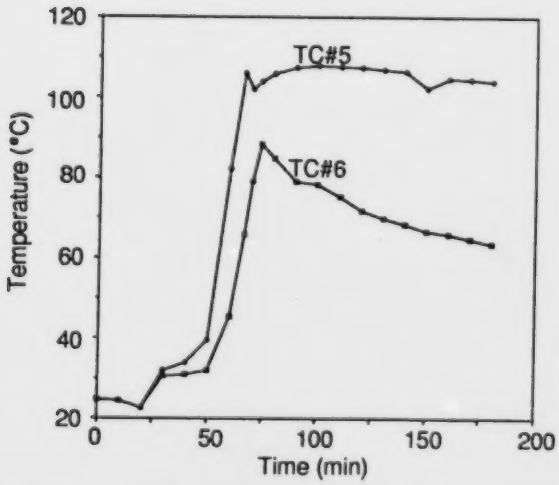

Figure 5. Temperature History During the Early Cure Phase of a 100 L Specimen (see text for details)

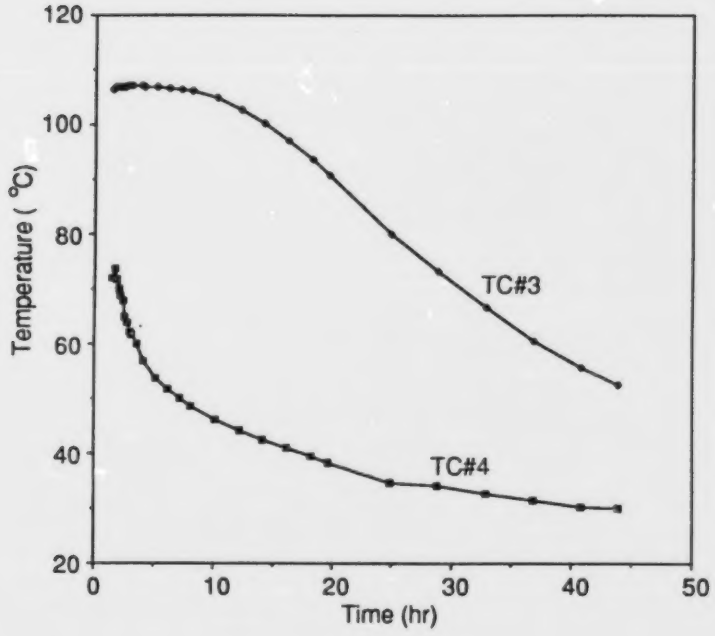

Figure 6. Temperature History During the Cure of a 100 L Specimen (see text for details)

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Photographs of the waste form shown in Figure 7 reveal cracks in both the top and bottom surfaces. These extended deep inside the solid and had volumes (estimated by filling with water) of ~1 and 0.7L, respectively. The split waste form, also shown in Figure 7, confirmed the deep penetration of the cracks and revealed the presence of additional minor ones. The flaws observed were probably the result of the large temperature gradients experienced by the solid. Note that no flaws were observed in any of the 20 L immobilized Calgon solution products which were also prepared using 4% catalyst. Presumably this may have been due to lower thermal gradients and hence lower thermal stresses in the smaller waste forms.

8.0 CONCLUSIONS AND RECOMMENDATIONS

The WEP immobilization plant has been fully commissioned. It performs satisfactorily under all its operational modes. Minor modifications to the equipment are needed to minimise operator intervention during use. An improved compact version of the equipment with appropriate shielding provisions would be suitable for field use. Due attention should be paid in the modified version to equipment decontaminability.

Immobilization experience with the WEP plant indicated that in general, conditions of neutral or close to neutral pH, a waste to resin volume ratio of unity and catalyst concentration up to 3% led to a uniformly cured solid waste form. Plant immobilizations must be based on bench scale solidifications to avoid adverse plant operating conditions.

Immobilizations to date have been performed using simulated decontamination solutions. Probable complications such as activity deposition due to processing of active waste solutions could not be assessed without relocating (to the Radioactive Materials Laboratory), making shielding provisions and possibly compacting (due to space constraints) the equipment. This assessment would be best made as part of field commissioning trials which would also indicate the extent of secondary waste arisings.

Additional experience in the preparation of large (greater than 20 L) waste forms is strongly recommended. Carbon steel containers (drums used in the field are made of carbon steel) should be used in these preparations to facilitate heat dissipation from within the solid and thus decrease the incidence of defects. Catalyst levels may also have to be lowered to facilitate the production of a defect-free solid even though this may extend the solidification period. These studies along with planned investigations on the effect of flaws on waste form acceptability would lead to a specification of the optimal size of waste forms.

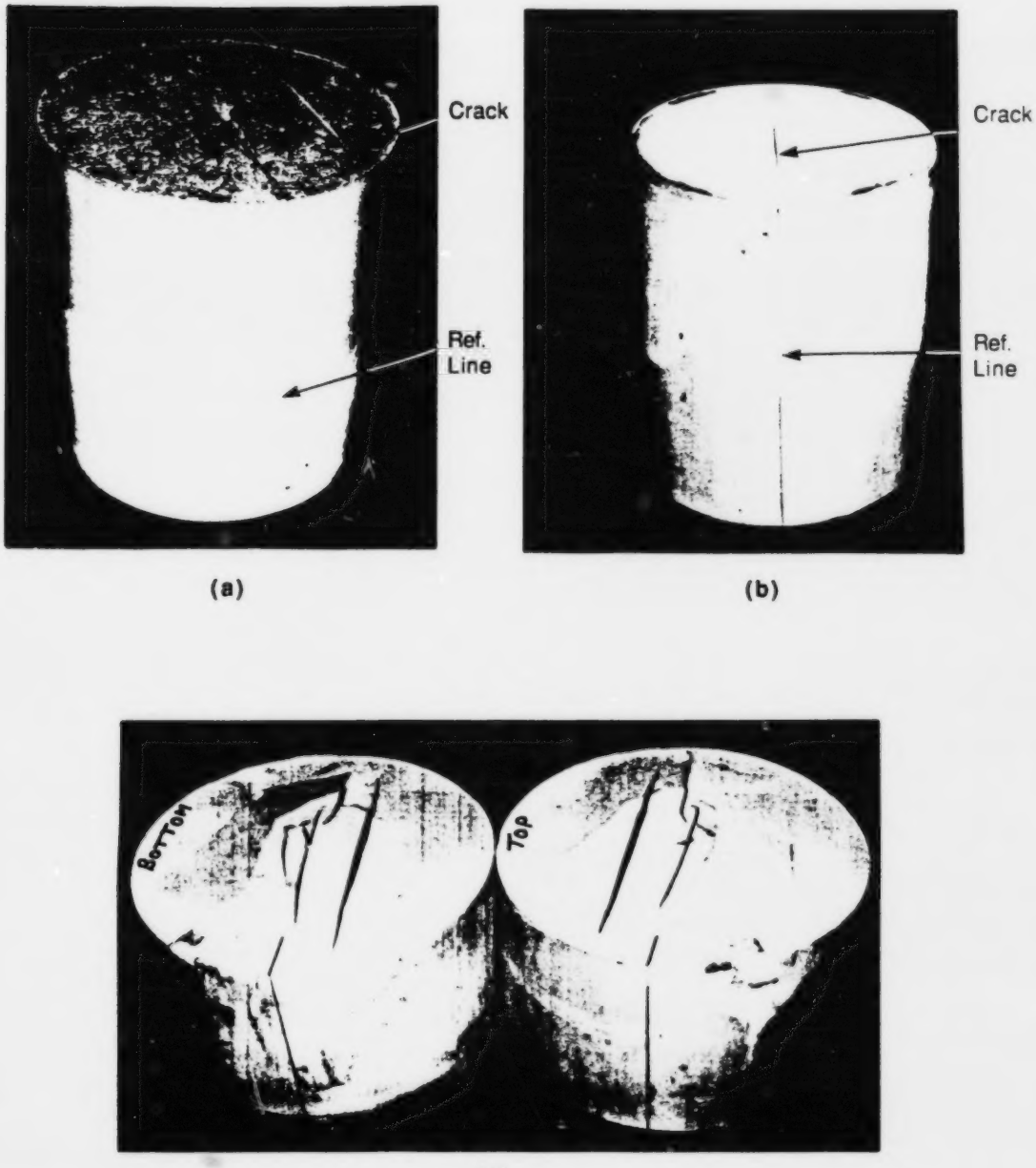

Figure 7. Photographs of the 100 L WEP Specimen Showing
a) its top surface
b) its bottom surface
c) sectioned halves

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9.0 ACKNOWLEDGEMENT

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APPENDIX

RELATIONSHIP BETWEEN FLOW RATE AND INTEGRATOR OUTPUT

A constant K can be defined such that the integrator output equals K x Flow Rate of Catalysed Emulsion x Filling time. The value of this constant was determined by observing the time to fill a container at a known flow rate until system shutdown occurred, i.e. when the accumulated pulses equaled the set count on the integrator. The value of K varied with the total flow rate; the variation is shown in Figure A.1. It was, however, independent of the integrator setting and the relative flow rates of emulsion, waste and catalyst.

The value of the constant can be used at any desired flow rate to determine the set count on the integrator for automatic shutdown of the WEP plant.

DELAY IN FLOW METER RESPONSE AND PUMP SEQUENCING

As described in Section 3.0, a zero flow condition in any (resin, waste or catalyst) line leads to system shutdown. This provision prevents start-up of the plant unless controller action is delayed sufficiently to allow for the finite response times of the flow meters. The latter were 1, 2 and 3s (averaged over a range of flow rates) respectively for the waste, catalyst and resin flow meters; these were determined by visually observing the flow indicators when the pump in each line was activated with the line filled between the pump and flow meter.

It was also established that under the usual range of flow rates, it would take about 5s after the initial start-up for the emulsion to enter the line blender. However, catalyst could be delivered to the line blender within 1s of the catalyst pump being activated. Therefore, if the catalyst flow was initiated 4s after start-up of the resin and waste flows, the emulsion entering the line blender would be appropriately catalysed. A more conservative approach was, however, adopted for sequencing the start-up of the pumps to ensure that emulsion is always catalysed. According to this the waste flow is activated 3s after both the resin and catalyst flows are simultaneously initiated.

The sequencing of the pumps and the ensuing flow meter responses are depicted in Figure A.2. Based on this, a delay of 5s was allowed for between the detection of a zero flow condition in any line and the subsequent shutdown of the plant.

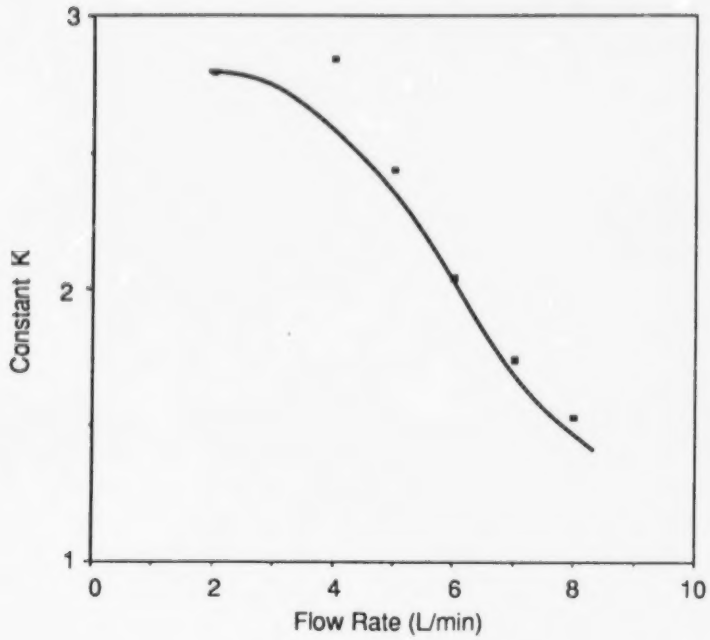

Figure A.1. The Flow Rate Dependence of Constant K

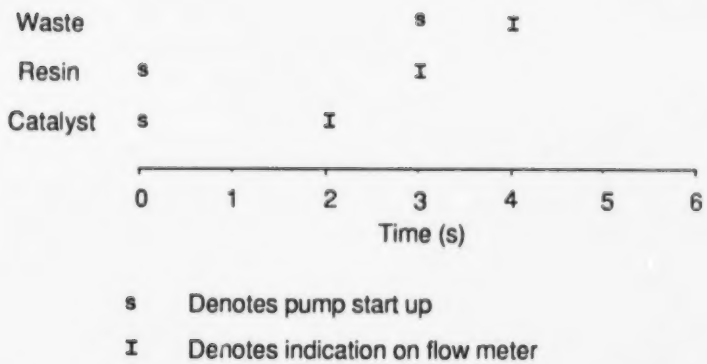

Figure A.2. Delay in Flowmeter Responses and Pump Sequencing